34

X-RAY STUDY OF CRYSTALLITE ORIENTATION IN AGAVE AMERICANA

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Plate VI

ABSTRACT. Following the method outlined by P. H. Hermans and others, a quantitative study of crystallite orientation in agave americana fibre under various physical conditions is carried out. It is found that unlike many other cellulose fibres, the orientation of the crystallites in agave americana shows improvement on treatment with NaOH. This is ascribed to the removal of some intercrystalline constituents.

Because of their textile importance cellulose fibres of different kinds and origin are probably the most extensively investigated of all the natural materials. They are known to occur as Natural, Bast and Leaf fibres. The determination of crystallite orientation in a particular type of leaf fibre-agave americana forms the subject matter of the present investigation

The problem of crystallite orientation in fibres is not only of theoretical interest but also of practical importance as many of their physical and chemical properties are correlated with it. A quantitative investigation of the problem has been attempted by Sisson and Clark (1933), Berkley (1939), R. Hosemann (1937) and Hermans and Hermans (1946) The problem has been studied both from X-ray and optical methods. The X-ray method is more useful because optical methods furnish information merely as to the average orientation of the entire fibrous substance, whereas the X-ray method gives orientation of only the crystallite components. It is in this restricted sense of orientation that a quantitative estimation by means of X-ray methods is made.

The intensity distribution along an X-ray interference circle from a polycrystalline specimen in which crystallites are oriented in some way, offers a possibility to investigate the orientation properties of the material. This was first pointed out and the foundations for such investigation were laid by Polanyi (1921). Since that time a very large number of orientation investigation has been published dealing with inorganic and organic materials of crystalline and semi-crystalline nature. Following Hermans, and Hermans, the orientation factor is determined by the equation

$$f_x = 1 - \frac{3}{2}\left(\overline{\sin^2\beta_1} + \overline{\sin^2\beta_2}\right) \qquad \ldots (1)$$

where β_1 and β_2 are the angular distances along the equator for two paratropic interferences arising from planes approximately at right angles to each other. In taking averages along the arcs we assign weights $G(\beta)\cos\beta_1$ and $G(\beta)\cos\beta_2$ the value of β according to the shape of the intensity distribution $I = G(\beta)$. Thus

$$\overline{\sin^2\beta} = \frac{\int I \sin^2\beta \cos\beta \, d\beta}{\int I \cos\beta \, d\beta}$$

However, since in those cases where $(10\bar{1})$, (021) overlap the intensity curve for $(10\bar{1})$ alone can not be determined, the following formula is used instead of (1)

$$f_x = 1.245 - 1.72 \sin^2\beta - 2.06 \overline{\sin^2 t}$$

where t is the angle along the $(10\bar{1})$—(021) circle relating to the total intensity of two overlapping interferences. This is based on K = 0.83

EXPERIMENTAL

Raw leaves of agave americana were retted as usual and the fibres obtained where thoroughly washed, dried and combed to ensure parallelism. Filtered CuK_α radiation from a Seifert's sealed tube working at 38 KV, and 18 mA was used with a specimen to film distance of 5 cm. and the specimen size, exposure time and photographic technique were standardised as far as possible. The photometer used was Moll's recording type The film was cut into a circular disc of 4.5 cm diameter and it was mounted on a rotatable holder fixed to the stage of the microphotometer so that the rotation which could be made in steps of 2° arc took place about the centre of the photograph. At each setting the film was scanned radially by traversing the holder. Since the films were of low photographic density it was assumed in the calculations that the X-ray intensity was proportional to blackening and was thus linearly related to the logarithm of the intensity of transmitted light. The results obtained are given in Table I.

TABLE I

Sample	$\sin^2\beta_1$	$\sin^2\beta_2$	f_x
Native Fibre	.041	.055	.85
When treated with 18%NaOH and dried without tension	.026	.0406	.90
Treated with 18% NaOH and dried under tension	.016	.014	.96

Native fibre boiled with 2%H_2SO_4 band mercerised with 18% NaOH = 0.75

DISCUSSION

Agave americana fibres are stiff, bright and comparatively thicker than jute in the native form. On treatment with caustic soda solution the strength

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PLATE VI

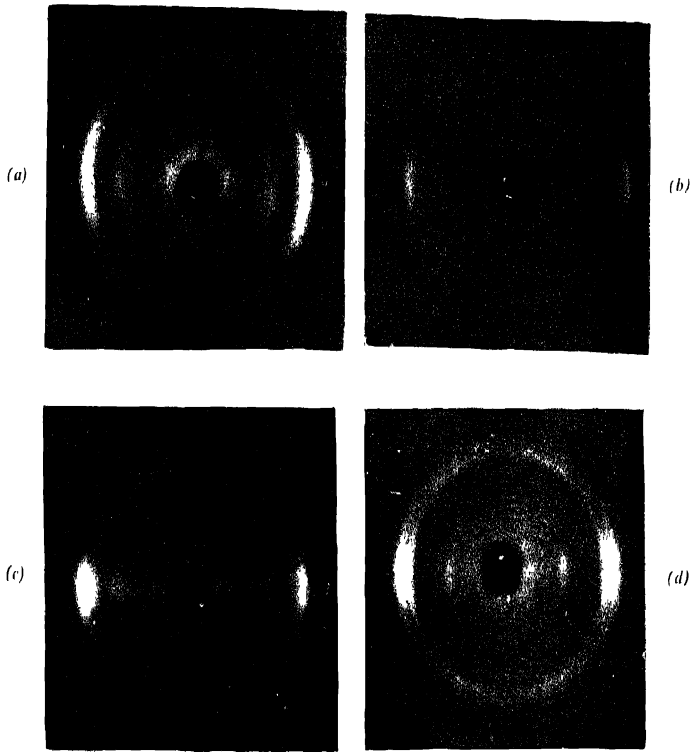

Fig. 1. X-ray photograph of the fibre

(a) Native fibre

(b) Treated with 17% NaOH, washed and dried without tension

(c) Same as above when dried under tension

(d) Boiled with 2% H_2SO_4 and then mercerised.

of the fibre gradually decreases which is evidently due to the removal of lignin and other intercrystalline materials which may also be responsible for an incomplete mercerisation. This is clearly seen from the X-ray photograph of the fibre treated with 18% NaOH (Plate VI). The photograph is a mixed one. The partial reconversion of the cellulose to the native modification can as well be ascribed to the formation of crosslinks between cellulose chains in the neighbourhood of the crystallites and thus creating a disposition in favour of the return to the original configuration after swelling. This seems to be supported by the fact that the degree of mercerisation is more pronounced in a sample which is boiled with 2% H_2SO_4 before being mercerised because it loosens the crosslinks. There is, however, also the possibility of the removal of some intercrystalline constituents which effect mercerisation. Another interesting observation made about agave americana is that the crystallites are better oriented as a result of the treatment with NaOH. This may again be due to the removal of those intercrystalline materials which impede the rotation and alignment of the crystallites. However when the fibre is first boiled with H_2SO_4 and then mercerised, the orientation decreases. This shows that the damage done on H_2SO_4 treatment far outweighs the improvement in orientation when NaOH alone is employed.

A detailed study of agave americana from the scattering of X-rays at small angles and by employing the techniques of electron microscopy is also being made and will be shortly reported.

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